PLASMODYNAMIC SYNTHESIS OF NANOSCALE SILICON CARBIDE: WAYS OF OPTIMIZATION

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Silicon carbide has been used in many fields of human activity: power electronics, the production of abrasive materials and ceramic products for working in corrosive environments. SiC is used due to its properties: high hardness, comparable with diamond, and wear resistance, wide band gap, refractory [1]. The characteristics of the materials can be improved by using nanoparticles in their production [2].

Silicon carbide synthesis techniques are not effective enough, because they have unsatisfactory dispersity, high duration etc. The synthesis of nano-sized SiC was carried out on a coaxial magnetoplasma accelerator (CMPA) [3]. This work is aimed at studying ways to optimize the plasmodynamic synthesis of nanoscale silicon carbide. Two series of experiments were carried out: changing the ratio of precursors and the influence of pressure in the reactor-chamber.

The identification of the best ratio of precursors is necessary to improve the purity of the product, to reduce the mass content of unreacted phases: silicon and carbon. Experiments with various precursors ratio (Si : C = 1.5 : 1, 2.3 : 1, 3 : 1, 9 : 1) were carried out. The intensity peaks on the X-ray diffractograms correspond to cubic silicon carbide. When the silicon content in the mixture is increased, the mass content of carbon in the synthesis product decreases.

Optimal phase composition (over 90 % silicon carbide) is achieved with a relative atomic ratio Si : C = 0.56 : 0.44 which corresponds to the mass ratio Si : C = 3 : 1. The stoichiometric equilibrium occurs when there is a lack of the carbon in the mixture, because carbon is partially produced by electroerosion from the graphite accelerating channel.

The synthesis and growth of silicon carbide crystals occurs in the head shock of plasma jet compaction. The reactor-chamber's atmosphere is located in the path of plasma propagation, thereby affecting the synthesis product.

A series with different pressures (low – 0.1 and 0.5 atm., normal – 1.0 and 1.5 atm., high – 3.0 and 5.0 atm.) was carried out to study the effect of the reactor-chamber's atmosphere pressure on the phase composition of the synthesized product.

The following dependence was revealed. The high pressure of atmosphere inhibits the propagation of plasma, thereby slowing the rate of the plasma flow. This means that the energy pT-parameters in the head shock of plasma jet compression decrease. Reducing energy parameters leads to decreasing formation of silicon carbide particles and increasing the content of unreacted silicon and carbon particles. At a low pressure, the lifetime of the quasistationary mode decreases significantly, that leads to a decrease in the content of cubic silicon carbide in the synthesized powder.

The result of this work is the optimization of the plasmadynamic synthesis of nanoscale cubic silicon carbide. The synthesis products were investigated by X-ray diffractometry (XRD) and transmission electron microscopy (TEM). Based on the results of X-ray diffraction analysis, it was found that the optimal phase composition (over 97 % in the synthesized product) is achieved when the rector-chamber's atmosphere pressure reaches p = 1.5 atm. and the precursors ratio reaches Si : C = 3 : 1. Transmission electron microscopy provides visual proofs of synthesis nanosized product (average particle size 70 nm).

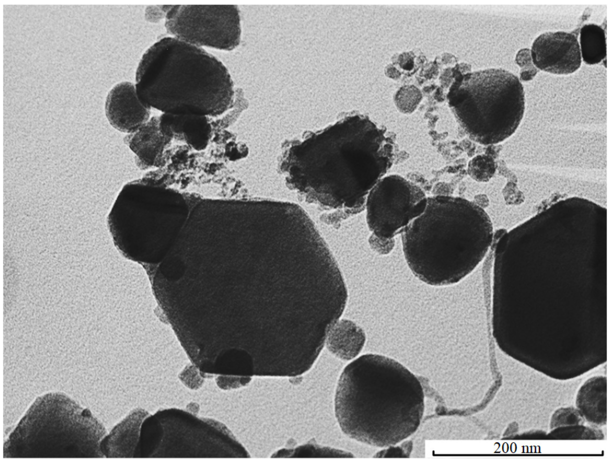

Fig. 1. *TEM-image of synthesized product*

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DEPENDENCE OF THE PRODUCT'S PHASE COMPOSITION ON THE RATIO OF PRECURSORS IN PLASMODYNAMIC SYNTHESIS OF TITANIUM DIBORIDE

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In present time materials with high physic-mechanical properties are in demand. Materials based on nanostructured powder show higher properties than coarse-grained analogs. Titanium diboride (TiB_2) is an excellent powder for functional ceramics, which needs in a lot of application in industry, for example, metallurgy, mechanical engineering [1]. It can be used as surface coatings on iron, steels, refractory metals, because TiB_2 has high hardness, good wear and corrosion resistance [2].

In this paper presents one of a possible way of obtaining TiB_2 – plasmodynamic synthesis using coaxial magnetoplasma accelerator (CMPA). The aim of this work was to find the optimal ratio of precursors to synthesize the phase of titanium diboride hexagonal syngony with spatial group P6/mmm. The possibility of synthesis nanopowders was shown in the work [3].

According to the aim series of experiments with different mass ratio of Ti:B precursors were implemented (26.7 : 73.3; 45.5 : 54.5; 61.1 : 38.9; 86.4 : 13.6).

Synthesized powder products without any pretreatment were analyzed by X-ray diffraction (XRD), transmission electron microscopy (TEM). Quantitative X-ray analysis was performed using the PowderCell program. The highest yield of the titanium diboride phase (96.2 %) was achieved in the experiment with a Ti : B ratio of 45.5 : 54.5, bur the average size of particles is 67.8 nm. The experiment with a boron percentage of 73 % was implemented, which from the Ti-B state diagram is the region for the production of titanium diboride. The yield of TiB_2 is 93.2 % and the particle size is the smallest in comparison with the other experiments – 56.1 nm.

The analysis of the crystal forms of the product was carried out on the basis of a set of light-field TEM and HRTEM images. There is typical bright-field image of transmission electron microscopy in figure 1. It can be seen that the product basically includes at least two types of particles. The first type is prismatic crystals of a hexagonal and dihexagonal forms, which looks like a circle in a plan. This form corresponds to the spatial group P6/mmm of the hexagonal system. The habit of a crystal can exist in several simple forms: hexagonal and dihexagonal prisms and pyramids. In other areas they can be related to the second type – crystals in the form of a cube that corresponds to the spatial group Fm3m for the corresponding phase of titanium diboride TiB cubic syngony. This product consists only of particles of two fractions: cubes and hexagons with a narrow width distribution up to 100 nm in size.

Thus, plasmodynamic synthesis of nanocrystalline particles of titanium diboride with hexagonal syngony with the spatial group P6/mmm was car-

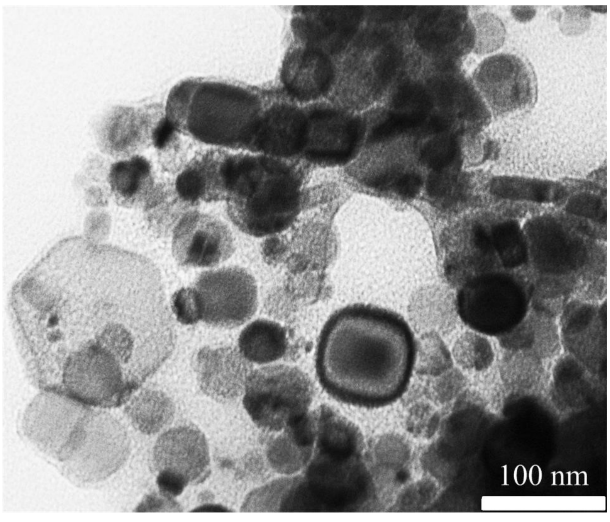

Fig. 1. *Typical bright-field image of transmission electron microscopy*